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CHAPTER

3

Fiber-top sensors

3.1 Optical fibers

Nowadays, optical fibers are used in a wide variety of applications. The main utilization is in data communication applications, which is an enormous market making these optical cables inexpensive and easily available. Because of their advantages compared to free space optics, they are also extensively used in scientific applications. Furthermore, they are extremely robust, flexible and available with many different specifications.

A typical optical fiber is mainly made out of glass and has a core with a slightly higher refractive index than the surrounding cladding region. As a result, light introduced at one end of the fiber is guided by total internal reflection (TIR) in the core and exits at the other end with very limited losses. To fabricate such a fiber, a glass rod is doped with a rare earth metal to increase the refractive index of the core and then heated and drawn into a thin fiber. As a protective layer, a polymeric coating is applied. In this way, many different kinds of fibers, supporting many kinds of applications, can be fabricated.

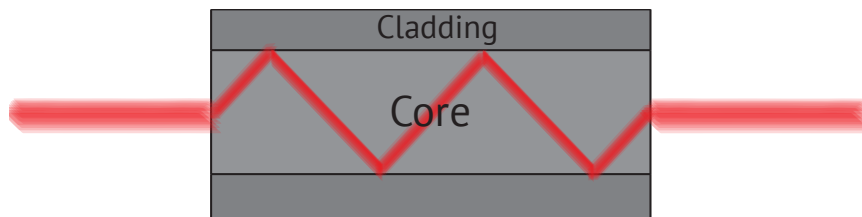


Figure 3.1: Schematic view of light guidance in an optical fiber by total internal reflection (TIR).

3.2 Fiber-top sensors

Optical fibers are primarily designed for light transportation, but can also be used to fabricate a variety of sensors on one of the end faces, which allows one to perform measurements in remote locations.

A cantilever is a very versatile type of force sensor that can be used in many applications, such as stiffness measurements, pressure sensing or in seismic observations. A small cantilever can be fabricated at the end face of a standard single mode fiber as demonstrated by Iannuzzi *et al.* in 2006 [1] (see Fig. 3.2). The deflection of this tiny cantilever can be detected by an interferometric readout at the proximal end of the fiber [2–7].

Fiber-top probes have been demonstrated to be of use in different applications, such as hydrogen sensing [3], temperature measurements [8] and atomic force mi-

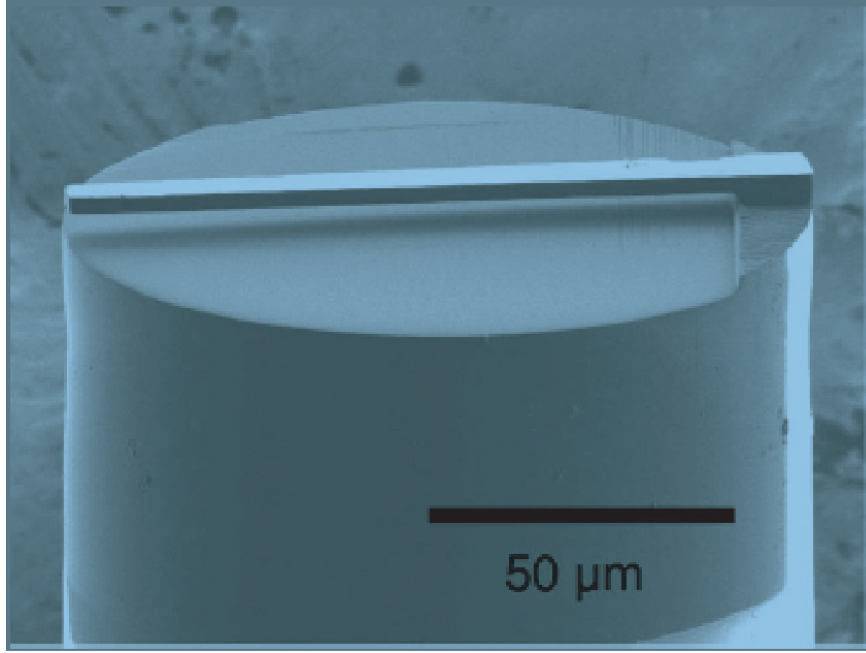


Figure 3.2: Illustration of a fiber top cantilever. Taken from Ref. [1].

croscopy [1, 5]. In addition, scanning near-field optical microscopy (SNOM) is another field where fiber-top probes can be applied. In this approach the excellent light guidance properties of fiber-optic cables are combined with the force sensing capabilities of fiber-top probes [9].

3.2.1 Interferometric detection

The deflection of the cantilever can be detected remotely by an interferometer connected to the proximal end of the fiber. As illustrated in Fig. 3.3, light reflects at the glass-to-air (W_1), air-to-glass (W_2) and glass-to-air (W_3) interfaces. The intensity of the interference signal W at the photodiode can be described by:

$$W = W_1 + W_2 + W_3 - 2\sqrt{W_1W_2}\cos\left(\frac{4\pi d}{\lambda}\right) - 2\sqrt{W_1W_3}\cos\left(\frac{4\pi d}{\lambda} + \frac{4\pi nt}{\lambda}\right) + 2\sqrt{W_2W_3}\cos\left(\frac{4\pi nt}{\lambda}\right) \quad (3.1)$$

where d is the gap between the fiber and the cantilever, t the thickness of the cantilever, n the refractive index of the fiber core, and λ the wavelength of the laser [1].

The exact values of W_1 , W_2 and W_3 are not known, therefore, Eq. 3.1 can be

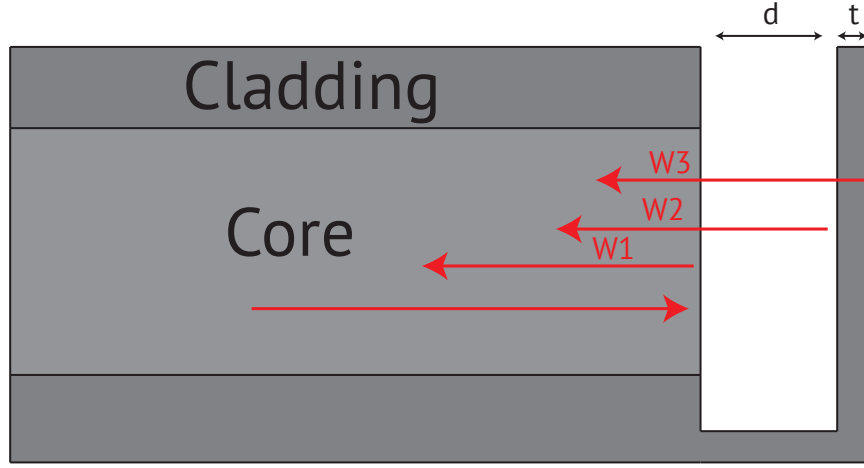


Figure 3.3: Schematic view of the interference-based feedback in a fiber-top device (not to scale).

written in a simpler form describing the relation between the cantilever deflection and the detected signal:

$$W = W_0 \left[1 - V \cos \left(\frac{4\pi d}{\lambda} + \phi \right) \right] \quad (3.2)$$

where W_0 is the midpoint interferometry signal, V the fringe visibility and ϕ a constant phase shift. W_0 and V are obtained from the maximum (W_{max}) and minimum (W_{min}) interference signals following:

$$V = \frac{W_{max} - W_{min}}{W_{max} + W_{min}} \quad (3.3)$$

and

$$W_0 = \frac{W_{max} + W_{min}}{2} \quad (3.4)$$

In the linear part of the interference fringe, the signal is most sensitive to cantilever displacement. Since the gap size d is fixed in these kind of sensors, the probe can be tuned into quadrature (midpoint interference) by changing the laser wavelength. The displacement sensitivity Δd can then be obtained from the minimum detectable signal ΔW [10]:

$$\Delta d \approx \frac{\lambda}{4\pi} \frac{\Delta W}{W_0} \frac{1}{V} \quad (3.5)$$

3.2.2 Fabrication of fiber-top probes

The diameter of a standard single mode fiber is 125 μm . A focused ion beam, integrated with a scanning electron microscope (FIB-SEM), can be used to fabricate tiny structures on top of such an optical fiber. A detailed description of the fabrication method can be found in Deladi et al. [2] and is illustrated in Fig. 3.4. In short, the fiber first needs to be coated with a metal to make it conductive and prevent charging, as this would make electron imaging impossible. After mounting the fiber in the FIB, a ridge is milled as shown in Fig. 3.4b. Subsequently, at the end of the ridge the two sides of a pyramid can be milled if a sharp tip is required for the application at hand (Fig. 3.4c). To finish the probe, it has to be rotated by 90 degrees. An undercut is made and the tip is finalized (Fig. 3.4d-f). Finally the remaining pole is removed to release the cantilever (Fig. 3.4g).

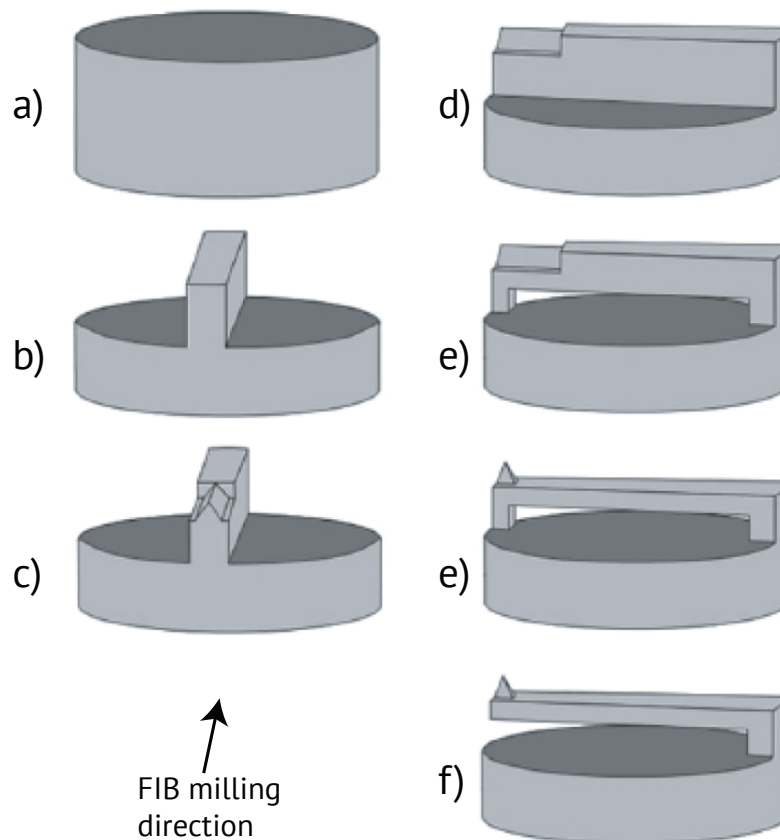


Figure 3.4: Illustration of the fabrication procedure of a fiber top probe. End face of a fiber (a), ridge formation (b), start of the sharp tip (c), fiber rotated in the FIB by 90°(d), undercut (e), tip finished (f), cantilever released (g). Taken from Ref. [2].

The small dimensions require a complicated, and expensive fabrication technique

that is difficult to automate. Attempts have been made to replace the FIB by a femtosecond laser micro-fabrication method [11]. This technique relies on the internal modification of the glass. The illuminated areas will etch much faster in hydrofluoric acid (HF) solution and can thus be removed by dipping the probe in a HF bath after the material modification. Laser machining is suitable for automation and is significantly cheaper than FIB. However, the resolution is poor and surfaces are rougher, which results in decreased performance of the resulting probe. Li *et al.* [8] showed that a combination of picosecond laser ablation and FIB can work. The main parts of the probe were machined using ps-laser ablation, while the cantilever was thinned and smoothened using the FIB.

A different fabrication approach would be photolithography. This technology is used in conventional MEMS fabrication and is therefore easily adapted to batch processing. In the Iannuzzi group, a method for batch fabrication of fiber-top cantilevers using photolithography was proposed by Petrusis *et al.* in 2009 [12]. In this method, known as Align-and-Shine, one mask fiber can be used to fabricate the same pattern on many different fibers. However, many different steps are required that need to be carefully optimized in order to be able to efficiently batch process many fibers. It may be a promising method to make the fabrication process more efficient in the near future.

3.3 Ferrule-top cantilevers

The fabrication process can be simplified by increasing the dimensions by one order of magnitude [13, 14]. Ferrule-top probes are very similar to fiber-top cantilevers and preserve many advantages [13–17]. These probes have been demonstrated to work for atomic force microscopy, as a photoacoustic spectrometer [18] and are especially suitable for force sensing of biological tissues [19–21].

3.3.1 Fabrication of ferrule-top probes

Ferrule-top probes consist of a single mode fiber terminated by a glass ferrule or rod containing a micro-cantilever. Borosilicate glass is a very robust and hard material, ideal for sensors that need to work in different environments such as liquids and at elevated temperatures.

The starting material of a ferrule-top probe is a borosilicate glass ferrule (dimensions: $3 \times 3 \times 7 \text{ mm}^3$). At one end of the ferrule a ridge is carved using a diamond wire cutter machine (Fig. 3.5a). A glass ribbon is glued to the ridge and cut to the right dimensions using picosecond laser ablation (Fig. 3.5b-c). Finally, a single

mode fiber can be inserted in the center bore hole to detect the cantilever deflection (Fig. 3.5, and a tip can be anchored to the end of the cantilever (Fig. 3.5e-f).

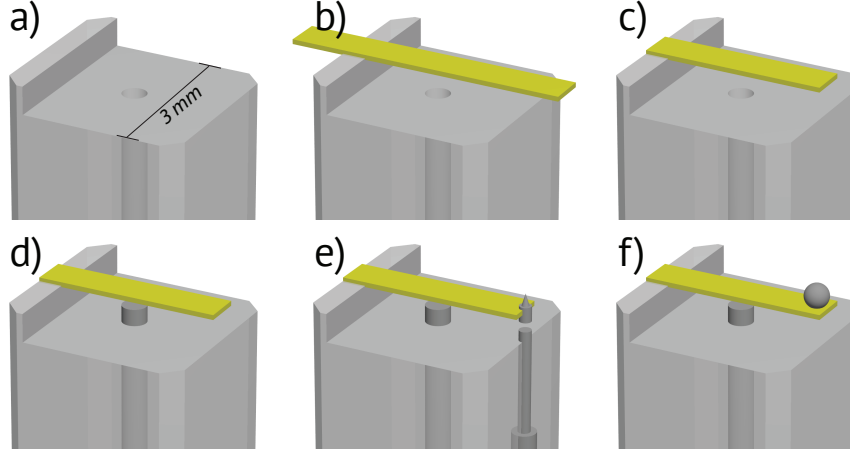


Figure 3.5: Required steps in the general fabrication procedure of ferrule-top probes. $3 \times 3 \times 7 \text{ mm}^3$ ferrule with a micro-machined ridge (a), a gold coated glass ribbon glued to the ridge (b), ferrule with cantilever (c), fiber for the interferometric readout inserted in the center bore-hole (d), probe containing a sharp tip (e) or a sphere at the end of the cantilever (f).

Depending on the application, the probe can be modified to fit the requirements. A sharp tip at the end of the cantilever allows for nanoscale surface analysis as in AFM imaging (Fig. 3.5e). A sphere or cylinder, on the other hand, is more appropriate for force sensing of soft tissues (Fig. 3.5f). The resulting stiffness of the cantilever can be approximated by the dimensions and material properties of the cantilever. The spring constant k is given by:

$$k = \frac{Ewt^3}{4L^3} \quad (3.6)$$

where E is the Young modulus of borosilicate glass (ca. 78 GPa), w the width, t the thickness and L the length of the cantilever. From this equation it is clear that the thickness and the length have a large effect on the stiffness of the rectangular beam. To tune the stiffness of the cantilever, and thus the force applied to a sample, the length, thickness and width can be varied.

3.3.2 Tip preparation

In order to make ferrule-top probes suitable for AFM imaging a sharp tip at the end of the cantilever is required. Since the probe dimensions are much larger than conventional micro-cantilevers used in AFM imaging, these probes are most suitable for contact mode imaging. Therefore, the tip shape is critical. To obtain nanometer

resolution images the sharpness is very important, but the tip also has to be very rigid to resist the relatively high contact forces.

Different tip fabrication methods known from the field of scanning near-field optical microscopy (SNOM) were described in section 2.4.4. The approach that we have applied for ferrule-top probe fabrication uses the difference in etching rate of the core and the cladding of the fiber, known as selective etching. Following the procedure described by Pangaribuan *et al.* [22], a special fiber with a high germanium doping concentration in the core is immersed in buffered HF solution with concentration 1 : 1 : 7 (HF : H₂O : NH₄F). The doping has a gradient profile in the core with the highest concentration in the center. Using this method, theoretically a tip size of 10 nm can be obtained [22]. Unfortunately, commercially available highly Ge-doped fibers have a dip in the concentration profile at the center, which limits the tip diameter to about 80 nm in practice.

3.4 Ferrule-top probes for optical surface analysis

Because ferrule-top probes are fully fiber connected, they can be constructed in such a way that the sharp tip can be used to transfer light to or from a sample. In other words, scanning near-field optical microscopy can be incorporated in an easy-to-use, plug-and-play probe. In fact, this will allow one to perform SNOM measurements in different environments, including liquids, and at elevated pressures and temperatures.

3.4.1 Fabrication of optical ferrule-top probes

The fabrication procedure is similar to the procedure used for standard devices as described in section 3.3.1. However, the placement of the imaging tip is more critical since the light transmission through the gap between the body of the probe and the tip needs to be as good as possible (see Fig. 3.6). This can be achieved by accurate alignment of the tipped fiber and by keeping the gap width below 5 μm . With perfectly smooth parallel surfaces the optical losses inside the gap can be calculated using [23]:

$$Loss = 1 - \frac{1}{Z^2 + 1} \quad (3.7)$$

in which Z represents:

$$Z = \frac{\lambda}{2\pi n_0 \omega_0} \cdot d \quad (3.8)$$

where λ is the wavelength, n_0 the refractive index of the environment outside the fiber, ω_0 the mode field radius and d the separation of the two fibers. A gap width of $5\ \mu\text{m}$ thus results in a loss of about 0.047 dB (ca. 1%). Misalignment is however much more sensitive to losses:

$$Loss = 1 - e^{-\left(\frac{x}{\omega_0}\right)^2} \quad (3.9)$$

where x represents the lateral misalignment.

As a starting point, a standard probe is fabricated according to the procedure described in Fig. 3.5a-d. To create the SNOM tip, a fiber is etched using the procedure described in section 3.3.2 to a final diameter of 20 to 30 μm . For robustness, it is important that the etched part is not longer than 5 mm. It is important that the strong polymeric fiber coating can be glued to the ferrule, as shown in Fig. 3.6b.

The basic probe is mounted in the laser ablation setup with the cantilever pointing upwards and a 40 μm wide groove is ablated over the length of the ferrule. Next, at the back of the probe the width of the groove is increased to about 300 μm to support the fiber coating of the etched fiber.

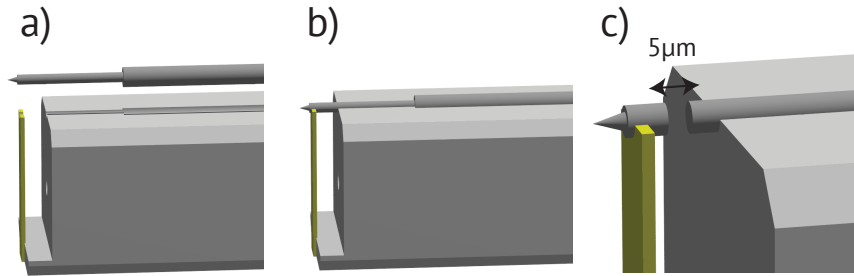


Figure 3.6: Fabrication procedure of ferrule-top AFM+SNOM probes. Alignment of the tipped fiber above the groove (a), gluing the tipped fiber inside the groove (b) and FIB cut between the probe body and the cantilever (c).

To mount the fiber tip on the ferrule, the fiber is aligned with the groove as shown in Fig. 3.6a under a microscope. When the fiber is exactly above the groove it is lowered in to the groove and glued in place (Fig. 3.6b). Finally, a tiny drop of glue is applied at the tip of the cantilever to fix the fiber tip. It is very important that the fiber is free of strain so that it will not move and misalign when it is being released. Before mounting the probe in the FIB-SEM, about 50 nm of aluminum is applied to the probe to increase the conductivity. The cantilever is then released by milling a 5 μm cut between the cantilever and the ferrule body using the FIB-SEM as illustrated in Fig. 3.6c. Maximum milling rate can be achieved by using a high FIB current, which allows one to finish the job in about 10 minutes. FIB milling produces very smooth surfaces and allows for optimal light transmission through the

gap. Finally, a small aperture is created at the tip apex using a very low FIB current to avoid damaging the tip.

3.4.2 Future work

FIB is an expensive technique, but it is an unavoidable tool in the current fabrication procedure of ferrule-top probes for optical surface analysis. In the current design, a precise cut between the tip and the rest of the fiber is required, which can only be done using the FIB. One could use laser ablation for this purpose and use heat to melt and smoothen the interfaces, but this is difficult to do reproducibly. Another challenge is the alignment; if the tipped fiber is under a little bit of stress it might misalign upon release of the cantilever and one needs to start over. Finding a different way of transmitting light to and from the sample would be beneficial for the applicability of these probes.

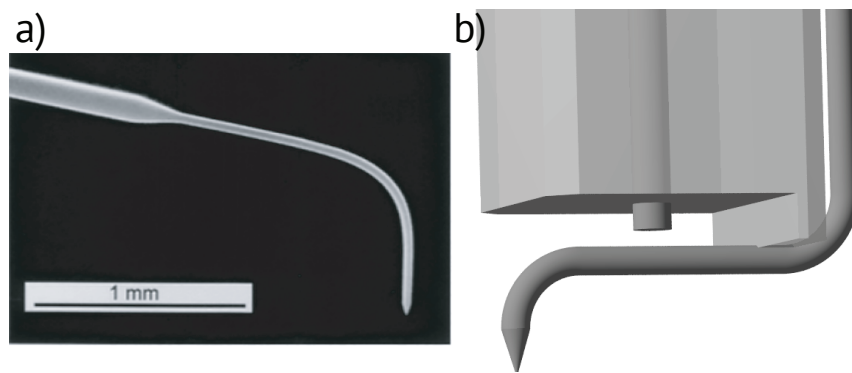


Figure 3.7: A bent fiber probe (a) (obtained from Muramatsu *et al.* [24]) and a schematic of a possible design using such a bent fiber as cantilever in a ferrule-top probe (b).

An alternative approach might be replacing the cantilever by a bent fiber probe, as used by various groups [24–26]. Incorporating such a probe in ferrule-top technology preserves the advantages, while FIB milling is avoided. A possible design, replacing the cantilever by a bent fiber probe, is proposed in Fig. 3.7b.

To fabricate a bent fiber probe, the tip can be created using the methods described before. Afterwards, the probe can be bent by local heating by either a CO₂ laser [24] or an electric arc [25]. High bending losses of the fiber can be a concern using this approach. Standard single mode fibers support a bending radius of 10 mm without considerable losses. Low loss fibers can be bent in an even smaller coil. In addition, the commercially available fiber probes through Nanonics (Nanonics Imaging Ltd., Jerusalem, Israel) have bending angles between 40 and 60 degrees and no major signal losses have been reported with these probes.

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